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Spectral Fingerprinting of Polycyclic Aromatic Hydrocarbons in High-Volume Ambient Air Samples by Constant Energy Synchronous Luminescence Spectroscopy

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16. ABSTRACT

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Spectral Fingerprinting of Polycyclic Aromatic Hydrocarbons in High-Volume Ambient Air Samples by Constant Energy Synchronous Luminescence Spectroscopy†

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■ A high-volume sampler fitted with a glass-fiber filter and backed by polyurethane foam (PUF) was employed to collect airborne particulate and gas-phase polycyclic aromatic hydrocarbons (PAHs) in ambient air. Samples were collected from four sources representing a range of environmental conditions: gasoline engine exhaust, diesel engine exhaust, air near a heavily traveled interstate site, and air from a moderately polluted urban site. Spectral fingerprints of the unseparated particulate and gas-phase samples were obtained by constant energy synchronous luminescence spectroscopy (CESLS). Five major PAHs in the gas-phase extracts were characterized and estimated. The compatibility of a high-volume sampling method using polyurethane foam coupled with CESLS detection is explored for use as a screening technique for PAHs in ambient air.

Introduction

Ambient air monitoring techniques are needed to provide rapid characterization and semiquantitation of pollutant levels of polycyclic aromatic hydrocarbons (PAHs) at different remote sampling sites (*1*). In this work, spectral fingerprints of PAHs in chromatographically unseparated particulate- and gas-phase air sample fractions were obtained by constant energy synchronous luminescence spectroscopy (CESLS). Combining CESLS with a high-volume air sampling technique offers (1) an inexpensive, simple, selective, and sensitive method for further application to remote site monitoring, (2) rapid collection of a large ambient air sample mass, and (3) fingerprints and semiquantitation of PAHs in gas-phase fractions containing two- to five-ring PAHs and fingerprints of particulate fractions containing five-ring and larger PAHs.

The amount of PAHs measured at a sampling data site is strongly influenced by sampling methodology (*2–20*). Conventional high-volume sampling of particulate organic matter with a glass-fiber filter is effective in collecting the five-ring and larger PAHs. The two- to five-ring PAHs associated with the particulate fraction tend to volatilize and pass through the glass-fiber filter as a result of the high flow rates and long sampling times. Yamasaki et al. (*3*) and Cautreels and Van Cauwenberghe (*4*) have studied the relationship between the particulate phase/gas-phase distribution factor and collection temperature for three- to six-ring PAHs. The three- to five-ring PAHs were found to be partitioned between the particulate and gas phases in proportion to the collection temperature. Thus, reliable characterization of PAHs (with less than six rings) in air samples requires the collection of both particulate and gas-phase fractions.

By using polyurethane foam plugs to back up the glass-fiber filters (GFF) during high-volume sampling, Thrane and Mikalsen (*6*) successfully collected both gas- and particulate-phase PAHs in ambient air.

Polymeric resins such as XAD-2, Chromosorb, and Tenax-GC are effective adsorbents for PAHs but cause high back-pressure in sampling systems operated at high air flow rates because of their finally divided, granular nature. The lower flow rates dictated by the polymeric sorbants have been offset by lengthening the collection period (order of weeks) in ambient air to provide a detectable PAH sample mass (*4*). Polyurethane foam (PUF) has been demonstrated to be an effective adsorbent for numerous semivolatile organic compounds even at relatively high air flow rates (*3, 6, 9–16*). Thus, a high-volume sampling technique combining glass-fiber filters with polyurethane foam (PUF) plugs was used in this study to collect both particulate-phase and gas-phase PAHs for evaluation by CESLS.

A predecessor to CESLS, constant wavelength synchronous luminescence spectrometry (CWSLS), was developed in the early (1970s by Lloyd (*21–23*) and has been used for numerous applications (*24–28*) discussed in detail by Vo-Dinh. Inman and Winefordner developed CESLS in 1983 (*29, 30*). During CESLS scanning, a constant energy difference is maintained between the two scanning (excitation and emission) monochromators. The constant energy difference is chosen to equal the prominent vibrational loss during an absorption–fluorescence transition (*29–31*). For the analysis of polycyclic aromatic hydrocarbons (PAHs) in the gas- and particulate-phase extracts the constant energy difference was chosen to equal the Stokes shift energy of 200 cm^{-1}, or the energy of the predominant –C=C– vibrational mode of 1400 cm^{-1}. The advantages of CESLS for multicomponent analysis are elimination of Rayleigh and Raman solvent scatter interferences and production of simple, reliable, reproducible spectra. These advantages have been discussed previously (*29–31*).

The main goal of this work was to evaluate the combination of high-volume sampling using PUF adsorbent with CESLS analysis as a potentially useful and simple screening technique for airborne PAHs. Filter extracts of gasoline engine exhaust, diesel engine exhaust, and ambient air were analyzed by CESLS. Major PAHs in the gas-phase fractions were semiquantitated.

Experimental Section

Sampling System. Samples were collected by using a standard General Metal Works (Cleveland, OH) high-volume air sampler modified by attaching a 7.5 cm × 16.5 cm glass cylinder to the motor inlet throat. The cylinder directed the flow of air from the glass-fiber filter through two polyurethane plugs. Two precleaned polyurethane plugs were slightly compressed and loaded into the glass cylinder for sampling. The sampling cylinder was elevated approximately 1 cm above the air inlet. The distance between the glass-fiber filter and the inlet to the polyurethane plugs was approximately 2 cm. The sampling apparatus was similar to two systems previously described (*3, 6*). A complete description of the modified high-volume

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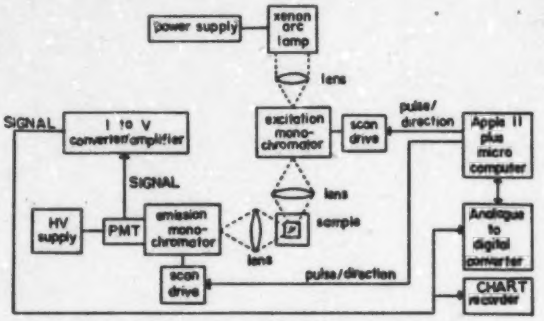

Figure 1. Schematic diagram of CESLS experimental setup.

sampler and sampling conditions for this study has been given (*7*).

Sample Collection. Andersen glass-fiber filters, 8 in. × 10 in., were used for collection of the particulate sample fraction, and polyurethane foam plugs, 7.5 cm o.d. × 8.5 cm, were used for collection of the gas-phase fraction. Polyurethane foam, type 3014 with a density of 0.022 g/cm^3, was supplied by Olympic Products Company, Greenboro, NC.

Prior to sample collection, the glass-fiber filters were fired for 4 h at 400 °C to remove any organic contaminants. The polyurethane foam cleanup and extraction procedures have been described by Erickson et al. (*16*), except that hexane was used instead of toluene as the solvent. The foam plugs were submerged in a beaker of hexane and further cleaned in a ultrasonic water bath. The hexane was decanted from the beaker, and the procedure was repeated.

After sampling, the glass-fiber filters were Soxhlet extracted for 24 h with 150 mL of hexane. The solvent extraction procedure for the polyurethane plugs was similar to the cleaning procedure excluding the ultrasonic agitation. The hexane extracts, 150 mL for the glass-fiber filter and 500 mL for the polyurethane plug, were reduced by a vacuum rotary evaporator to final volumes between 3 and 7.5 mL.

Sampling Methodology. Four air samples were collected: (1) gasoline engine exhaust from a four-cylinder, 1983 Honda Civic; (2) diesel engine exhaust from a four-cylinder, 1982 Volkswagen Rabbit; (3) ambient air in the vicinity of heavily traveled interstate I-95, in Jacksonville, FL; (4) ambient air from the rooftop of Black Hall, University of Florida Environmental Science and Engineering Building, in an area that is moderately urbanized. Only one sample was collected in each case as the study was exploratory in nature.

For the collection of the automobile exhausts, the high-volume sampler was modified with a 6 in. o.d. × 3 ft length of stovepipe to direct engine exhaust to the sampler inlet and allow partial cooling of the exhaust. Exhaust samples were collected for 20 min while the automobiles were idling. Ambient air samples were collected over a 48-h period with the sampler mounted in a conventional protective housing. The Black Hall sample was collected approximately 40 ft above the ground. The Jacksonville air sample was collected at a permanent ambient air monitoring site with the sampler located approximately 10 ft above the ground. Sampling conditions are listed in Table I.

CESLS Instrumentation. The experimental setup used for this work is shown in Figure 1. The equipment is listed in Table II. An Apple II Plus microcomputer controlled the simultaneous excitation–emission monochromator pulsing. The scan rate was 100 nm/min. to attain a constant energy synchronous scan, the excitation monochromator scanner was driven at a constant rate over the wavelength range of 200–500 nm, and the emission scanner was driven at a variable, faster rate determined by values from a "lookup" table. Spectral band-passes were 4 and 8 nm. Spectra were not corrected for instrument response. The sample cell was a Suprasil quartz cuvette, 1.2 cm × 1.2 cm × 4.5 cm. A Keithley current amplifier with a time constant of 300 ms was employed.

Table I. Experimental Components for CESLS

equipment	model	manufacturer
xenon arc lamp, 150 W	VIX-150 UV	EIMAC, Division of Varian, San Carlos, CA 94070
illuminator power supply, operated at 12 A	P250S-2	EIMAC, Division of Varian, San Carlos, CA 94070
excitation monochromator f/3.5, holographic grating 1200 grooves/mm	H-10 UV	American ISA, Inc., Metachen, NJ 08840
emission monochromator f/3.5, holographic grating 122 grooves/mm	H-10 V	American ISA, Inc., Metachen, NJ 08840
monochromator scan controls	1020-SS	American ISA, Inc., Metachen, NJ 08840
photomultiplier	1P928	Hamamatsu, Waltham, MA 02154
high-voltage power supply		laboratory constructed
microcomputer	Apple II Plus	Apple Computer, Inc., Cupertino, CA 95014
recorder	Omni Scribe	Houston Instrument, Austin, TX 78753
current amplifier	427	Keithley Instruments, Inc., Cleveland, OH 44139

Table II. Physical Sampling Conditions for Collection of Four Environmental Samples

sample	high-volume sampling rate, m^3/h[a]	sampling duration, h	total volume sampled, m^3
diesel exhaust[b]	47	0.33	16
gasoline exhaust[b]	27	0.33	9
Black Hall air	32	48	1520
Jacksonville air	70	48	3360

[a] Air flow rates are at 24 °C. [b] Collection temperatures at the glass-fiber filter surface were 60 °C.

CESLS Methodology. Spectral fingerprints were obtained by CESLS at constant energy differences equal to the Stokes shift energy ($\simeq$200 cm^{-1}), to one vibrational quantum ($\simeq$1400 cm^{-1}), and to three vibrational quantum ($\simeq$4800 cm^{-1}). In order to eliminate Raman interferences from hexane, a constant energy difference of two vibrational quanta ($\sim$3000 cm^{-1}) was not chosen for CESLS scanning. Hexane, used in the extraction process, has a Raman band of 3000 cm^{-1}. Prior to spectral analysis, the PUF extracts were diluted 1:100 with hexane, except for the Black Hall sample which was diluted 1:10. The GFF extracts were diluted 1:100 with hexane. Stock solutions of PAHs were made with spectroscopic-grade hexane obtained from Burdick & Jackson and from Fisher Scientific. Aliquots of the PAH solutions were used for standard addition and semiquantitation of the PUF extracts. Sources of the PAHs have been listed previously (31).

Results and Discussion

CESLS Spectral Fingerprinting. Because PAHs undergo strong absorption–fluorescence transitions, CESLS has proven to be a viable technique for PAH

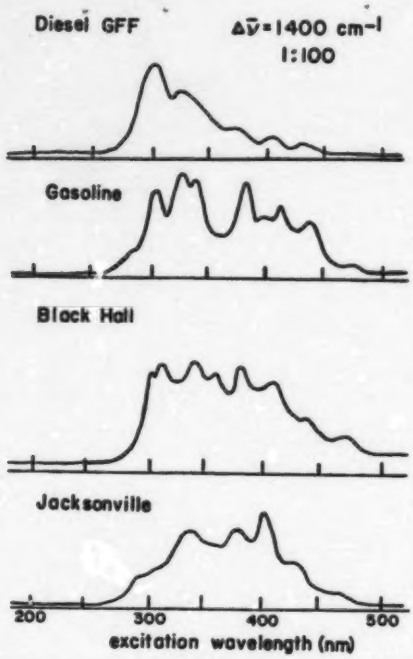

Figure 2. CESLS (at $\Delta\bar{\nu} = 1400$ cm^{-1}) of four glass-fiber filter extracts diluted 1:100 with hexane.

analysis (*29–31*). CESLS was chosen as a spectral fingerprinting technique because (1) CESLS scanning reduces spectral complexity and minimizes Raman scatter interferences observed in conventional fluorescence scanning and (2) CESLS gives analytical figures of merit for PAHs comparable with those obtained by fluorescence measurements at excitation and emission wavelength maxima (*31*).

Spectral fingerprints were obtained on the filter extracts with CESLS scanning at $\Delta\bar{\nu} = 200$, 1400, and 4800 cm^{-1}. The filter extracts were analyzed by CESLS without prior separation. The excitation, emission wavelength pairs corresponding to the $\Delta\bar{\nu}$ values listed previously (*31*) were used as a guide for the initial fingerprint evaluation. All 17 PAHs listed had excitation, emission wavelength pairs with a $\Delta\bar{\nu}$ of $\simeq$4800 cm^{-1}. Twelve of the 17 PAHs had wavelength pairs with $\Delta\bar{\nu}$ of $\simeq$1400 cm^{-1}. Only six PAHs, anthracene, benzo[*k*]fluoranthene, benzo[*a*]pyrene, benzo[*ghi*]perylene, fluorene, and perylene, had wavelength pairs with $\Delta\bar{\nu} \simeq 200$ cm^{-1}. At $\Delta\bar{\nu} = 4800$ cm^{-1}, all PAHs could be spectrally determined, but the scans were of greater complexity than with the 1400-cm^{-1} scans or the 200-cm^{-1} scans. By scanning with the three $\Delta\bar{\nu}$ values, the presence of PAHs could be cross-checked. The 1400-cm^{-1} scans aided greatly in the characterization of the major PAHs present in the PUF extracts.

GFF Extracts. The CESLS scans of the four GFF extracts are shown in Figure 2. The 1400-cm^{-1} scan illustrates the spectral complexity of the GFF extracts. Little spectral information was obtained from the 200-, 1400- or the 4800-cm^{-1} scans due to the spectral complexity of the GFF extracts. The diesel and gasoline engine exhausts and the Black Hall spectra show a peak at an excitation wavelength of 300 nm. All four spectra have peaks at approximately 330, 380, 410, and 435 nm of varying intensities. Besides the spectral similarities noted, few qualitative conclusions can be drawn concerning the PAH composition of the unseparated (or "crude") particulate extracts of the automobile exhausts and the ambient air samples.

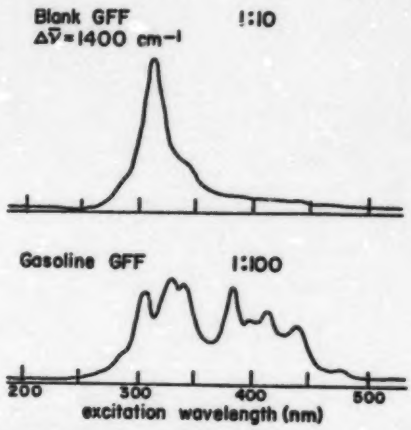

Figure 3. Comparison of hexane-rinsed glass fiber filter extract (1:10 dilution) to gasoline exhaust glass fiber filter extract (1:100 dilution) by CESLS at $\Delta\bar{\nu} = 1400$ cm^{-1}.

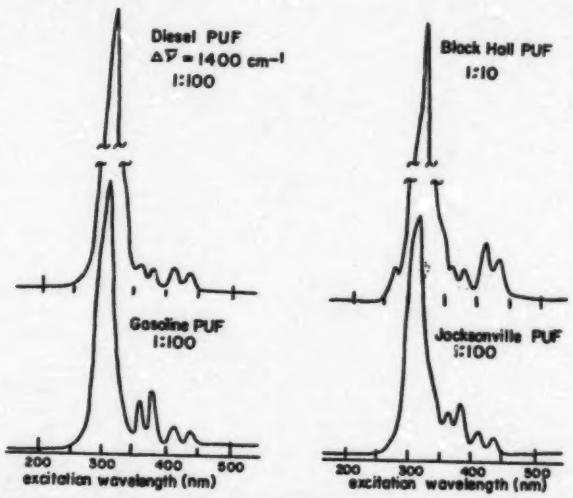

Figure 4. CESLS at $\Delta\bar{\nu} = 1400$ cm^{-1} of four polyurethane foam extracts diluted 1:100 with hexane (except Black Hall extract diluted 1:10).

The CESLS scan of the GFF blank is shown in comparison to the gasoline GFF scan in Figure 3. The blank was obtained by rinsing the GFF filter with hexane. The blank extract was diluted 1:10 before scanning. A single peak is seen at 310 nm with a shoulder at approximately 335 nm. The contaminant in the GFF blank was not identified and appeared to present little spectral interference in the scans of the four GFF samples.

PUF Extracts. The spectral finterprints of the PUF extracts at $\Delta\bar{\nu} = 1400$ cm^{-1} are shown in Figure 4. There is a large reduction in spectral complexity over the glass fiber filter scans. This reduced complexity is likely the result of a physical separation process, as the retention of organic vapors on PUF during high-volume sampling is selective for volatile and semivolatile compounds of relatively high molecular weights (*33*). All four extract fingerprints have similar peaks at excitation wavelengths of 300, 360, 380, 410, and 440 nm. Variations in the spectral intensities of these peaks occur in each fingerprint.

The PUF blanks were prepared by successive hexane rinsings of the foam plugs and were concentrated from 500 to 30 mL. Spectral analysis of the first washing of 1:100 dilution is shown in Figure 5. An intense peak at 300 nm was observed. The second washing was obtained on the same foam plug, and the 300-nm peak was reduced by a

Table III. Semiquantitation of Five PAHs Found in Four Polyurethane Foam Extracts by CESLS

sample	phenanthrene, $\mu g/m^3$[a]	anthracene, $\mu g/m^3$	fluoranthene, $\mu g/m^3$	fluorene, $\mu g/m^3$	pyrene, $\mu g/m^3$
diesel exhaust	6.4	0.4	0.2	4.9	0.4
gasoline exhaust	2.1	0.6	0.3	4.3	0.3
Black Hall air	0.01	0.001	0.0009	0.006	0.0009
Jacksonville air	0.02	0.006	0.01	0.01	0.008

[a] Amount of each PAH per volume of air sampled, $\mu g/m^3$, was determined by standard addition, $\mu g/mL \times$ original volume $\times$ 1/volume of air sampled. The original volume for all four samples was 20 mL. The volume of air sampled in each collection has been listed in Table II.

Table IV. Mass Spectrometry Analysis of Five PAHs Found in Polyurethane Foam Extracts

sample	phenanthrene, $\mu g/m^3$[a]	anthracene, $\mu g/m^3$	fluoranthene, $\mu g/m^3$	fluorene, $\mu g/m^3$	pyrene, $\mu g/m^3$
diesel exhaust	3.6	0.3		2.7	
gasoline exhaust	3.8	1.1		7.5	
Jacksonville air	0.01	0.003	0.007	0.003	0.004

[a] The amount of each PAH, in $\mu g/mL$, was determined by ratioing the peak areas of the 50 $\mu g/mL$ standard and the samples. The amount of each PAH per volume of our sample, $\mu g/m^3$, was determined by standard addition, $\mu g/mL \times$ original volume $\times$ 1/volume of air sampled. The original volume for all four samples was 20 mL. The volume of air sampled in each collection has been listed in Table II.

factor of 2. The contaminant spike was identified as butylated hydroxytoluene (BHT), a widely used preservative and antioxidant, added in many foam-making processes.

Semiquantitative Analysis. In order to characterize and quantitatively estimate the PAHs in the PUF extracts, spectral information from each sample at 200, 1400, and 4800 cm^{-1} was compared to a PAH standard spectra obtained at the three energy differences. From the literature (*2–4, 32, 33*) and previous work (*31*), the major PAHs found in gas-phase automobile exhaust were tabulated. As a result five PAHs were selected for mixture analysis: anthracene, fluorene, phenanthrene, fluoranthene, and pyrene.

From the 200-cm^{-1} scans, spectral information about anthracene and fluorene could be obtained. With the 1400-cm^{-1} scans and through the use of standard addition, anthracene, fluorene, and pyrene were characterized and semiquantitatively determined. With the 4800-cm^{-1} scans and standard addition, the five PAHs were all characterized and estimated. Mixtures of PAH standards were made up for the PUF samples. Figure 6 demonstrates the "fit" of the standard PAH gasoline mixture spectrum to the gasoline exhaust spectrum at $\Delta\bar{\nu} = 1400\ cm^{-1}$. The standard mixture has been shaded in. The semiquantitative values are listed in Table III (in $\mu g/m^3$) of air sampled. The detection limits for the five PAHs at $\Delta\bar{\nu} = 4800\ cm^{-1}$ have been determined previously (*31*). The detection limit is defined as the amount giving a signal 3 times the standard deviation of 20 blank measurements. For the five PAHs, the detection limits are the following: (1) anthracene, 9 pg; (2) fluorene, 5 pg; (3) fluoranthene, 18 pg; (4) phenanthrene, 180 pg; (5) pyrene, 36 pg (*31*).

To compare the analytical results obtained by CESLS, PAH standards and PUF extracts (except Black Hall sample) were analyzed by gas chromatography/mass spectrometry (GC/MS; Model 5985 B, Hewlett Packard, Palo Alto, CA). PAH standards (Foxboro Analabs, North Haven, CO) were prepared in hexane and consisted of 100 $\mu g/mL$ fluorene and 50 $\mu g/mL$ each of phenanthrene, anthracene, fluoranthene, pyrene, triphenylene, benz[*a*]anthracene, chrysene, benzo[*e*]pyrene, perylene, and benzo[*a*]pyrene.

GC/MS analysis was performed by a 2-μL splitless injection (30-s septum purge interrupt) onto a fused silica capillary column (0.25 mm × 30 m DB-5; J & W Scientific, Inc., Rancho Cordova, CA). After a 2-min hold at 60 °C, the column was temperature programmed to 280 °C at 10 °C/min. The mass spectrometer was operated in the electron ionization mode (70 eV; source temperature 100 °C) and was scanned from 45 to 500 amu. Peak areas were determined from extracted ion current profiles of the molecular ion for each PAH. The PAH concentrations in the PUF extracts were calculated by ratioing the peak areas of the standard and the sample. The CESLS results of Table III compared favorably to the mass spectrometry results in Table IV by a factor of 0.6–2.

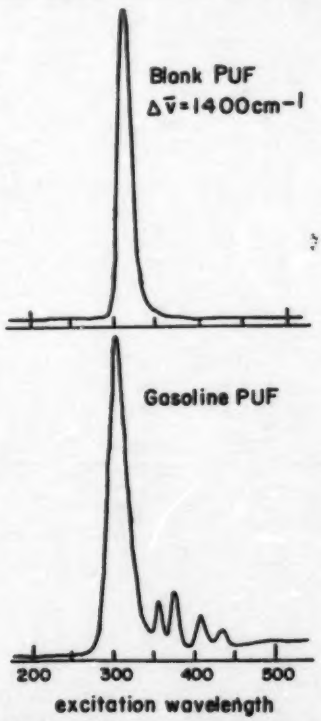

Figure 5. Comparison of hexane-rinsed polyurethane foam extract to gasoline exhaust polyurethane foam extract by CESLS at $\Delta\bar{\nu} = 1400\ cm^{-1}$. Both samples are diluted 1:100 with hexane.

Since only four samples were collected in this preliminary study and sampling efficiencies were not evaluated, the semiquantitative values listed in Table III are, by no means, indicative of the concentrations found in all diesel and gasoline exhausts. The feasibility of CESLS fingerprinting as a screening technique for the presence of major

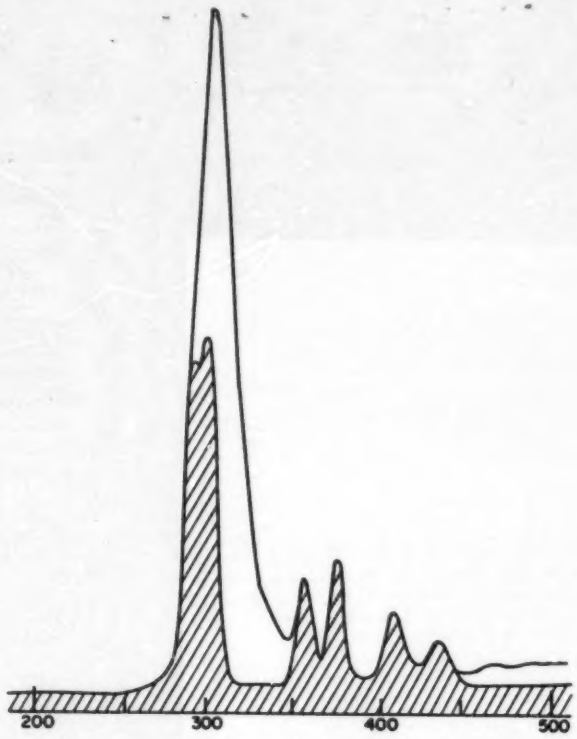

Figure 6. CESLS at $\Delta\tilde{\nu}$ = 1400 cm^{-1} of gasoline exhaust polyurethane foam a mixture of five PAH standards (shaded in) to demonstrate spectral similarities.

PAHs in air samples ranging in concentration and composition from automobile exhaust to ambient air was demonstrated in this study by the analysis of chromatographically unseparated filter extracts. Furthermore, CESLS analyses of preseparated GFF and PUF filter extracts could be used to provide increased detail and quantitative information on sample composition. The combination of a high-volume sampling technique, using polyurethane foam adsorbent and glass-fiber filters for gas- and particulate-phase PAH collection, with CESLS analysis is proposed as a simple, inexpensive, sensitive, and specific method for characterization and semiquantitation of airborne PAHs.

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Registry No. Anthracene, 120-12-7; fluorene, 86-73-7; fluoranthene, 206-44-0; phenanthrene, 85-01-8; pyrene, 129-00-0.

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